



Novel ternary molten salt electrolytes for intermediate-temperature sodium/nickel chloride batteries

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HIGHLIGHTS

- ▶ Ternary low melting point catholytes were developed.
- ▶ New catholytes revealed lower melting temperatures and adequate properties for the Zebra battery.
- ▶ The cell with the new catholyte exhibited reduced polarizations at 175 °C compared to the standard catholyte.
- ▶ The battery revealed stable cycling performance at temperature as low as 150 °C.

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ABSTRACT

The sodium–nickel chloride (ZEBRA) battery is operated at relatively high temperature (250–350 °C) to achieve adequate electrochemical performance. Reducing the operating temperature in the range of 150–200 °C can not only lead to enhanced cycle life by suppressing temperature-related degradations, but also allow the use of lower cost materials for construction. To achieve adequate electrochemical performance at lower operating temperatures, reduction in ohmic losses is required, including the reduced ohmic resistance of β'' -alumina solid electrolyte (BASE) and the incorporation of low melting point secondary electrolytes. In present work, planar-type Na/NiCl₂ cells with a thin BASE (600 μ m) and low melting point secondary electrolyte were evaluated at reduced temperatures. Molten salts used as secondary electrolytes were fabricated by the partial replacement of NaCl in the standard secondary electrolyte (NaAlCl₄) with other lower melting point alkali metal salts such as NaBr, LiCl, and LiBr. Electrochemical characterization of these ternary molten salts demonstrated improved ionic conductivity and sufficient electrochemical window at reduced temperatures. Furthermore, Na/NiCl₂ cells with 50 mol% NaBr-containing secondary electrolyte exhibited reduced polarizations at 175 °C compared to the cell with the standard NaAlCl₄ catholyte. The cells also exhibited stable cycling performance even at 150 °C.

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1. Introduction

Sodium–metal chloride batteries, referred to as the Zebra battery, were initially developed for electric vehicle (EV) application [1–5]. They have several advantages over Na/S batteries including lower operating temperatures, improved safety, higher voltage, easiness of assembly in discharged state, and better tolerance against overcharging [6–9]. Recently, the Zebra battery has gained more attention as an energy storage device for renewable energy applications [6,7].

Among the several types of Zebra batteries, most widely investigated has been nickel-based chemistry, which is typically fabricated in a tubular form with a β'' -alumina solid electrolyte (BASE) tube [10]. Cathode materials typically consist of electrochemically active ingredients (nickel and sodium chloride in discharged state) and a molten salt secondary electrolyte (or catholyte) such as NaAlCl₄, which ensures facile sodium ion transport between the BASE and active cathode materials. A small amount of additives such as NaF, FeS, and Al are also added to the cathode to minimize the degradation of battery performance caused by overcharge abuse, grain growth of nickel, and sudden polarization drop at the end of discharge [4,10–12]. Since this battery is assembled in discharged state, no anode material (sodium metal) is added on assembly. Molten sodium forms at the

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anode during the first charge. The normal cell reactions during discharge are as follows [10]:

Anode: $\text{Na} \rightarrow \text{Na}^+ + \text{e}^-$

Cathode: $\text{NiCl}_2 + 2\text{Na}^+ + 2\text{e}^- \rightarrow \text{Ni} + 2\text{NaCl}$

Overall: $2\text{Na} + \text{NiCl}_2 \rightarrow 2\text{NaCl} + \text{Ni}$, $E_0 = 2.58 \text{ V}$ at 300°C .

The ZEBRA battery is usually operated at relatively high temperatures ($250\text{--}350^\circ\text{C}$), which is well above the melting point of the liquid secondary electrolyte (NaAlCl_4 ; $T_m = 157^\circ\text{C}$), in order to achieve adequate battery performance by reducing the ohmic resistance of thick BASE ($1\text{--}2 \text{ mm}$ thick) and improving the ionic conductivity of the secondary electrolyte [1,10]. Since previous results indicate that particle growth and side reactions occurring in the cathode are also enhanced at high operating temperatures, lowering the operating temperature ($\leq 200^\circ\text{C}$) with maintaining performance capability is beneficial to improve the cycle life of the Zebra battery [10,13,14]. The reduced operating temperature may also allow for low cost materials of construction and high throughput manufacturing methods, providing a path towards broad market penetration for grid storage. In order to decrease the operating temperature as low as 150°C , two important issues need to be addressed: (i) lowering the resistance of BASE and (ii) developing a low melting point catholyte with high ionic conductivity. To lower the resistance of BASE, a thin planar high-strength BASE ($300\text{--}600 \mu\text{m}$ thick), which is a composite of yttria-stabilized zirconia (YSZ) and β' -alumina, has been developed [15–17].

In the present work, the synthesis of novel low melting point catholyte was attempted by partially replacing NaCl ($T_m = 801^\circ\text{C}$) in the standard NaAlCl_4 with lower melting point alkali salts such as NaBr ($T_m = 747^\circ\text{C}$), LiCl ($T_m = 605^\circ\text{C}$), LiBr ($T_m = 505^\circ\text{C}$) since those salts are known to possess weaker ionic bond strength than NaCl [18]. The melting temperature, ionic conductivity and electrochemical window of partially NaCl-replaced secondary electrolytes will be reported in this paper. The intermediate-temperature electrochemical performance of the planar-type Na/NiCl₂ battery incorporating the low melting point catholyte will be also discussed.

2. Experimental

2.1. Materials and synthesis

To lower the melting temperature of secondary electrolytes, NaCl in NaAlCl_4 was partially replaced ($0\text{--}75 \text{ mol}\%$ replacement) with NaBr ($T_m = 747^\circ\text{C}$), LiCl ($T_m = 605^\circ\text{C}$), and LiBr ($T_m = 505^\circ\text{C}$), which have lower melting points than NaCl ($T_m = 801^\circ\text{C}$). High-purity alkali metal salts (99.99%) and anhydrous AlCl_3 (99.99%) procured from Alfa Aesar were used to synthesize ternary catholytes, following a method reported in the literature [19]. Alkali metal salts (a mixture of NaCl and another salt) and AlCl_3 were mixed in the molar ratio of 1.15 to 1 and homogenized at 320°C in a three neck flask which was purged with ultra-high purity (UHP) argon. An excess of alkali metal salts was employed to prevent the formation of Lewis-acid melts whose molar ratio of alkali metals to Al is less than 1 [19]. High purity aluminium foils were added during the homogenization to remove possible impurities. Elemental analysis confirmed that the level of impurities was less than 5 ppm. The melting temperature of as-synthesized secondary electrolytes was measured using a capillary melting point analyzer (Mettler MP50) in the temperature range between 80 and 200°C at the heating rate of 3°C min^{-1} . The nomenclature and composition

of each synthesized catholyte is listed in Table 1. The corresponding mole fraction of the salt substituted for NaCl is also shown.

2.2. Ionic conductivity and electrochemical window measurements

Measurements of ionic conductivity and electrochemical window were conducted in an argon-filled glove box (MBRAUN LABmaster SP/DP). The ionic conductivity of a molten catholyte was measured using an impedance analyzer (Solartron 1260A equipped with 1287A potentiostat) in the frequency range of 1 MHz to 0.05 Hz . The impedance measurements were performed at a series of temperatures from 150°C to 250°C using a two-probe method. The probe was made of two platinum foils ($3 \text{ mm} \times 3 \text{ mm}$) which were glass sealed on a rectangular alumina rod. Each probe was calibrated using standard solutions with three different concentrations (1 M , 0.1 M , and 0.01 M KCl aqueous solutions) to obtain accurate conductivities.

The electrochemical window of a secondary electrolyte was measured in a three-electrode cell using a potentiostat (Solartron 1287A). An molybdenum wire (0.5 mm OD) and foil ($5 \text{ mm} \times 10 \text{ mm}$) was used as working and counter electrodes, while an aluminium wire emerged in a borosilicate glass tube filled with an AlCl_3 -saturated $[\text{EMIM}]^+\text{Cl}^-$ solution was used as a reference electrode. Cyclic voltammograms were collected at the scan rate of 50 mV s^{-1} between 0 and 2.8 V with respect to the Al/Al^{3+} reference electrode.

2.3. Battery assembly and tests

Planar Na/NiCl₂ cells were assembled in a glove box, following the procedure described in our previous publication [6,7]. The assembled cell was initially charged up to 2.8 V at 280°C to obtain the full theoretical capacity ($\sim 150 \text{ mA h}$) at the constant current of 10 mA and discharged back to 80% of the initial maiden charge capacity. The cell was then cooled down to 175°C and 150°C and cycled between 20 and 80% state of charge (SOC) at $C/10$ (9 mA). The voltage limits of 2.8 and 1.8 V were applied to avoid overcharging and overdischarging, respectively.

3. Results and discussion

3.1. Characteristics of new secondary electrolytes

Fig. 1 shows the melting temperatures of NaAlCl_4 and various molten salt electrolytes obtained by partially replacing NaCl in NaAlCl_4 with lower melting temperature alkali metal salts. The melting point of secondary electrolytes containing NaBr decreases with increase in the amount of NaBr (158°C for NaAlCl_4 and 140°C for $75 \text{ mol}\%$ replacement). The molar ratio of $[\text{Br}^-]/[\text{Cl}^-]$ in the $\text{NaCl}/\text{NaBr}/\text{AlCl}_3$ system corresponds to 0.23 for $75 \text{ mol}\%$ replacement of NaCl (NaBr-75). This trend of melting point observed in the NaBr-substituted catholytes is in good agreement with the results of the $\text{NaAlCl}_4\text{--NaAlBr}_4$ system [20], which has a eutectic point at 142°C with $[\text{Br}^-]/[\text{Cl}^-] = 0.25$. Lowering melting temperatures by

Table 1
The nomenclature and composition of secondary electrolytes.

Salt	25 mol% replacement	50 mol% replacement	75 mol% replacement
NaBr	NaBr-25 ($\text{NaBr}_{0.25}\text{NaCl}_{0.75}\text{AlCl}_3$)	NaBr-50 ($\text{NaBr}_{0.5}\text{NaCl}_{0.5}\text{AlCl}_3$)	NaBr-75 ($\text{NaBr}_{0.75}\text{NaCl}_{0.25}\text{AlCl}_3$)
LiCl	LiCl-25 ($\text{LiCl}_{0.25}\text{NaCl}_{0.75}\text{AlCl}_3$)	LiCl-50 ($\text{LiCl}_{0.5}\text{NaCl}_{0.5}\text{AlCl}_3$)	LiCl-75 ($\text{LiCl}_{0.75}\text{NaCl}_{0.25}\text{AlCl}_3$)
LiBr	LiBr-25 ($\text{LiBr}_{0.25}\text{NaCl}_{0.75}\text{AlCl}_3$)	LiBr-50 ($\text{LiBr}_{0.5}\text{NaCl}_{0.5}\text{AlCl}_3$)	LiBr-75 ($\text{LiBr}_{0.75}\text{NaCl}_{0.25}\text{AlCl}_3$)

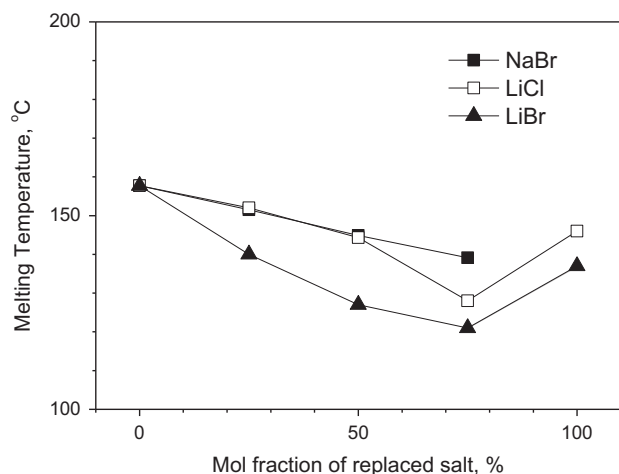


Fig. 1. Melting temperatures of secondary electrolytes measured using the capillary tube method. NaCl in NaAlCl₄ was partially replaced with NaBr, LiCl, and LiBr.

partial replacement of NaCl was also observed in NaCl/LiCl/AlCl₃ and NaCl/LiBr/AlCl₃ systems. It was generally understood that the melting temperature of molten salts is related to ionic bond strength. For example, NaBr ($T_m = 747$ °C) has a lower melting temperature compared to NaCl ($T_m = 801$ °C), since NaBr has weaker ionic bond strength due to the larger ionic radius of Br⁻ compared to Cl⁻. In the case of LiCl ($T_m = 605$ °C), ionic bond strength is weaker than NaCl even though the Li⁺ ion is smaller than Na⁺ ion. For LiCl, the electrons in Cl⁻ ions are pulled to the Li⁺ nuclei so that a more covalent nature is created compared to NaCl, weakening ionic bond strength [18]. Thus, the reduced polarity of Li⁺ ions compared to Na⁺ ions leads to a lower melting temperature of LiCl. These effects of Br⁻ and Li⁺ ions on ionic bond strength seem to result in the lower melting temperatures of the catholytes in which NaCl was partially replaced with NaBr, LiCl and LiBr.

The effects of NaCl replacement with lower melting point alkali metal salts on ionic conductivity are shown in Fig. 2. At the temperature of 175 °C or higher, all three catholyte systems (NaCl/NaBr/AlCl₃, NaCl/LiCl/AlCl₃ and NaCl/LiBr/AlCl₃) generally have similar or higher ionic conductivity than pure NaAlCl₄. The improved ionic conductivities of the NaCl/NaBr/AlCl₃, NaCl/LiCl/AlCl₃ and NaCl/LiBr/AlCl₃ can be attributed to its lower melting temperatures (low bond polarity) and more irregular structures of molten salts allowing easier ion hopping. The positive effects of NaCl replacement on the ionic conductivity are most obvious at 150 °C at which NaAlCl₄ exists as a solid. As shown in Fig. 2(b), NaCl-replaced secondary electrolytes still exhibit good ionic conductivity at 150 °C except one containing 25 mol% NaBr (NaBr-25). However, the ionic conductivity observed in this study may not necessarily represent the Na⁺ conductivity. The deviation between the total ionic conductivity and the Na⁺ conductivity can be more pronounced in the systems containing a higher fraction of Li salts due to a lower Na⁺ concentration.

The electrochemical windows of 50 mol% NaCl-replaced secondary electrolytes measured at 190 °C are shown in Fig. 3. It is known that the low voltage limit of NaAlCl₄ is set by the reduction of Al³⁺ (occurring at 0 V vs. Al/Al³⁺; standard reduction potential = -1.66 V) while the high voltage limit is restricted by the oxidation of Cl⁻. As can be seen, the low voltage limit of various secondary electrolytes was not changed since no alternation in AlCl₃ composition was made. However, the change in the high voltage limit was observed from the secondary electrolytes with NaBr and LiBr. This is due to the lower oxidation potential of Br⁻

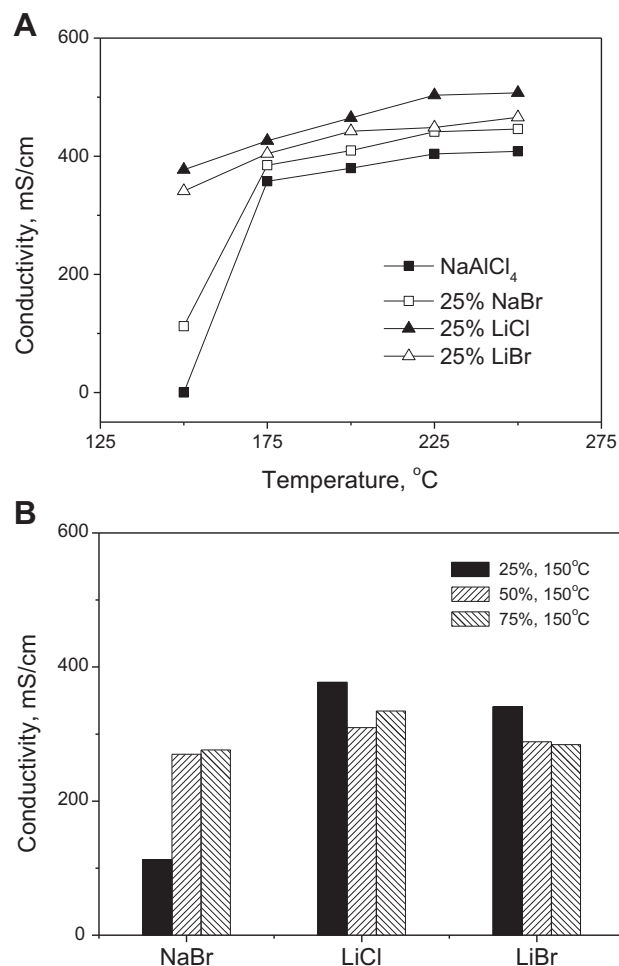


Fig. 2. Ionic conductivity of various secondary electrolytes: (a) ionic conductivity of NaAlCl₄ and 25 mol% replaced secondary electrolytes as a function of temperature and (b) ionic conductivity of secondary electrolytes with 25, 50 and 75 mol% NaCl replacement at 150 °C.

(standard reduction potential = 1.07 V, 2.73 V vs. Al/Al³⁺) compared to that of Cl⁻ (standard reduction potential = 1.36 V, 3.02 V vs. Al/Al³⁺). The high voltage limits of all the secondary electrolytes, however, are high enough to apply these catholytes for the Na/NiCl₂

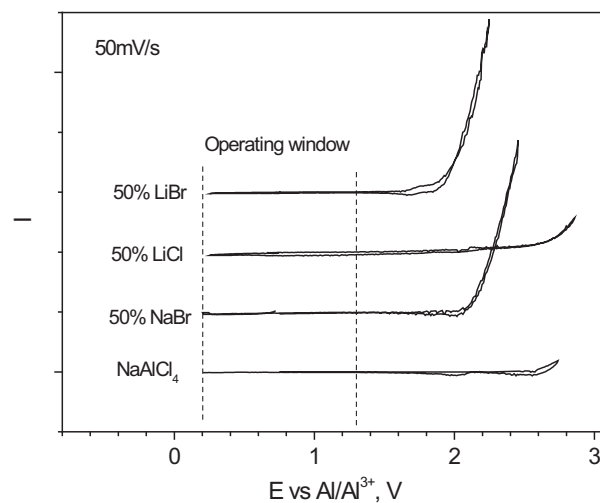


Fig. 3. Cyclic voltammograms of NaAlCl₄ and 50 mol% replaced secondary electrolytes measured at 190 °C.

batteries which cycle between 1.8 V (0.2 V vs. Al/Al^{3+}) and 2.8 V (1.2 V vs. Al/Al^{3+}) with respect to the Na/Na^+ potential.

3.2. Electrochemical performance

Na/NiCl_2 cells with one of the low melting temperature catholytes (NaBr-50: 50 mol% NaCl-replaced with NaBr) were tested

and compared with a cell containing a standard NaAlCl_4 secondary electrolyte.

The charge/discharge profile of the NaBr-50 cell is compared with the standard NaAlCl_4 cell in Fig. 4. At 280 °C, the cell with the NaBr-50 catholyte exhibited slightly smaller polarization (or lower cell potential) during charge and similar polarization during discharge (see Fig. 4a). The reduced polarization due to the use of lower melting temperature secondary electrolyte (NaBr-50) is more obvious at 175 °C as shown in Fig. 4b. The rapid polarization increase at the end of discharge (represented by a sharp drop in voltage) was also significantly reduced compared to the standard NaAlCl_4 cell. This result implies that the sharp drop in voltage at the end of discharge at 175 °C is related to not only the poor wetting of molten sodium to the BASE [14] but also the diffusion limitation of Na^+ ions in the secondary electrolyte, which is caused by the high viscosity of NaAlCl_4 at the low temperature close to its melting point. The cell with the NaBr-50 secondary electrolyte was able to be cycled even at 150 °C, where the standard NaAlCl_4 cell could not be cycled due to the high melting point of NaAlCl_4 ($T_m = 157$ °C). Only a limited capacity of 80 mA h (between 20% and 73% SOC) was cycled at 150 °C due to a rapid increase in cell voltage at the end of charge (refer to Fig. 4c). This rapid increase in voltage occurring only at 73% SOC implies that Na^+ ion conduction in the secondary electrolyte becomes a rate limiting step especially at the end of charge where the electrochemical reaction occurs farther from the cathode/BASE interface. The sharp drop of the cell potential at the end of discharge was also much more severe at 150 °C compared to 175 °C (Fig. 4c).

Fig. 5 shows the impedance spectra of the cells with the NaBr-50 catholyte compared with the standard NaAlCl_4 cell. In all the cells, slightly lower ohmic resistance (high-frequency intercept: HFI) was observed at the end of discharge (EOD) compared to the end of charge (EOC). This is due to the formation of an electrically less conductive NiCl_2 layer over Ni particles during the charge process. At 175 °C, a significant decrease in ohmic resistance was detected in the cell containing the NaBr-50 catholyte (1.08 Ω at EOC) compared to the standard NaAlCl_4 cell (1.49 Ω at EOC). The ohmic resistance of the NaBr-50 cell increased at 150 °C to 1.5 Ω at EOC, but it is still comparable to that of the standard NaAlCl_4 cell at 175 °C. Even though exhibiting similar ohmic resistance, the NaBr-50 cell tested at 150 °C revealed larger polarization arcs compared the standard

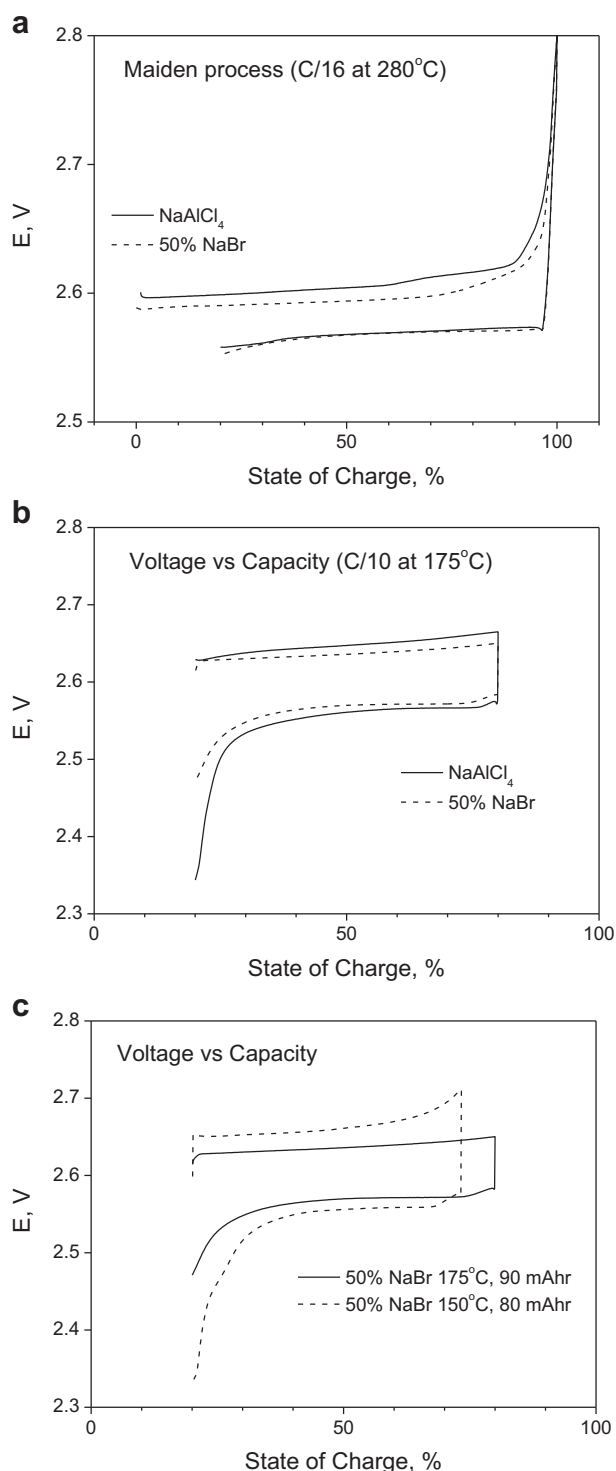


Fig. 4. Plots of charge–discharge voltage vs. the state of charge (SOC): (a) at 280 °C [maiden charge and discharge down to 20% SOC], (b) at 175 °C [cycled between 20 and 80% SOC], and (c) at 150 °C [only 80 mA h was cycled due to the voltage limitation of charge].

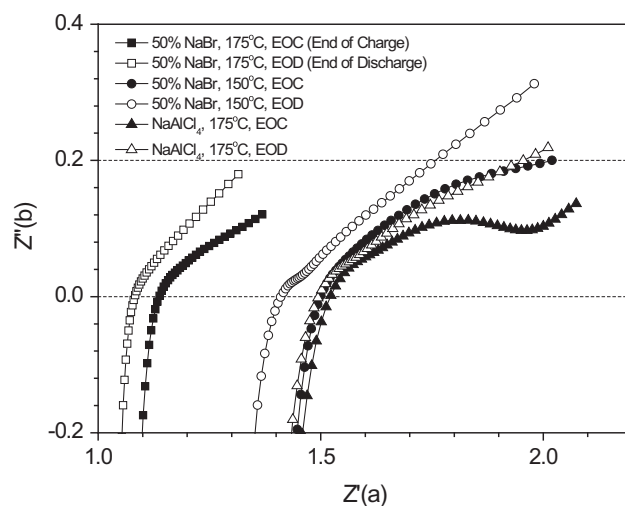


Fig. 5. Impedance spectra of cells containing NaAlCl_4 and the NaBr-50 secondary electrolyte. Impedance measurements were conducted at the end of charge (EOC) and at the end of discharge (EOD).

Table 2Ohmic resistances and total cell polarizations of the Na/NiCl₂ cell with the NaBr-50 catholyte at 175 °C.

	BOC ^a		EOC ^a		BOD ^a		EOD ^a	
Catholyte	NaAlCl ₄	NaBr-50	NaAlCl ₄	NaBr-50	NaAlCl ₄	NaBr-50	NaAlCl ₄	NaBr-50
Ohmic resistance (Ω)	1.49	1.08	1.52	1.14	1.52	1.14	1.49	1.08
Total cell polarization (Ω)	5.3	5.3	9.4	7.8	1.6	1.2	26.2	12.2

^a BOC: beginning of charge, EOC: end of charge, BOD: beginning of discharge, and EOD: end of discharge.

NaAlCl₄ cell tested at 175 °C, indicating the sluggish kinetics of electrochemical reactions. Since impedance spectra did not provide complete semicircles (or low-frequency intercepts), the total cell polarization was calculated from the difference between cell potentials at the end of each step and open circuit voltage (OCV). The total cell polarizations at the end of each step and the ohmic resistance obtained from the impedance measurements are listed in Table 2.

At the beginning of charge (BOC) and discharge (BOD), the electrochemical reactions (Ni oxidation for charging and NiCl₂ reduction for discharging) occur near the cathode/BASE interface. Therefore, the polarizations related to charge transfer and diffusion at BOC and BOD are much smaller compared to those at the end of the charge (EOC) and discharge (EOD) since the electrochemical reactions occur far from the cathode/BASE interface at the end of

each step. It is also observed that the total cell polarizations at BOC and EOD are larger than those at BOD and EOC even though the ohmic resistance is smaller. It should be noted that the cell is in discharged state in case of BOC and EOD, while it is in charged state for BOD and EOC. At temperatures lower than 200 °C, sodium melt at the anode reveals poor wetting to the BASE. Therefore, the polarization associated with poor sodium wetting is maximized in discharged state, where the least amount of sodium melt is left during cycling.

The cell performance of the battery with the NaBr-50 catholyte at 150 °C is shown in Fig. 6. No capacity degradation (Fig. 6a) or no significant change in end-of-step voltage (Fig. 6b) is observed for 50 cycles at the C/9 rate (9 mA). Overall, the stable performance of the NaBr-50 cell indicates that this secondary electrolyte is chemically stable without experiencing ion exchange of Br[−] in the catholyte with Cl[−] in the active cathode materials such as NaCl and NiCl₂. In the case that Br[−]–Cl[−] ion exchange occurred, the melting temperature and the viscosity of the catholyte would have increased with time so that the polarization should have increased with cycling.

4. Conclusions

Novel low melting temperature secondary electrolytes were developed by partially replacing NaCl in the standard NaAlCl₄ catholyte with other alkali metal salts such as NaBr, LiCl, and LiBr, which have lower melting points compared to NaCl. Partially NaCl-replaced catholytes revealed lower melting temperatures, improved ionic conductivity, and sufficient electrochemical window for the Na/NiCl₂ Zebra battery. The Na/NiCl₂ cell with the 50 mol% NaBr containing secondary electrolyte exhibited reduced polarizations at 175 °C compared to the cell with the standard NaAlCl₄ catholyte. The cell with NaBr-containing catholyte also exhibited stable performance at 150 °C, which is significantly lower than normal Zebra battery operating temperature (~300 °C). These result demonstrated the feasibility of intermediate-temperature Zebra batteries containing low melting point ternary catholytes.

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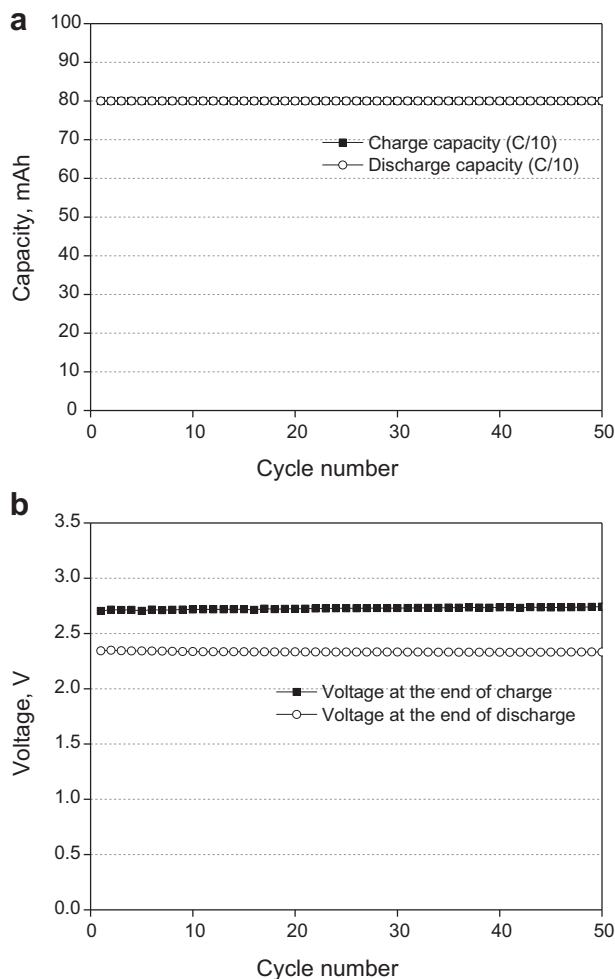


Fig. 6. Electrochemical performance of the cell containing the NaBr-50 secondary electrolyte at 150 °C: (a) capacity vs. cycle and (b) end-of-step voltage vs. cycle. The cycling capacity was 80 mA h [53% of theoretical capacity].

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